

Title of the Invention:

ZINC BORATE, METHOD OF PREPARING THE SAME AND

5 (Technical Field)

The present invention relates to a novel zinc borate, a method of preparing the same and use thereof. More specifically, the invention relates to a novel zinc borate having a particular crystallite size and containing very little sodium components and to a method of preparing the same.

(Background Art)

In recent years, resin molded articles have been used in a wide variety of fields, and it has been desired to impart flame-retarding property to these resins. It has further been desired that the resin molded articles such as building materials produce smoke in suppressed amounts so will not to hinder people from taking refuge in case fire has broken.

20 It has long been known that a zinc borate exhibits excellent flame-retarding property. For example, Japanese Examined Patent Publication (Kokoku) No. 67363/1981 teaches blending a zinc borate with a metal oxide such as Al₂O₃, SiO₂, Sb₂O₃, ZnO or ZrO₂.

Japanese Unexamined Patent Publication (Kokai) No. 137988/1988 filed by the present applicant discloses a flame-retarding composition obtained by treating the surfaces of a zinc borate particles with an oxide, a hydroxide or a basic carbonate of an alkaline earth metal of an amount of 1 to 40% by weight per the whole weight.

A method of preparing a zinc borate has also been long known. For example, Comparative Example 1 of Japanese Examined Patent Publication (Kokoku) No. 20902/1971 teaches the preparation of a compound of the

35 formula,

2ZnO·3B₂O₃·9H₂O

commercial value.

by mixing a combination of borax pentahydrate and boric acid with a zinc oxide in an aqueous solution of sulfuric acid, followed by the addition of seed crystals of a zinc borate.

However, the conventional zinc borate assumes the form of particles of indefinite shapes having relatively large diameters in which fine primary particles are coagulated in a random fashion and densely, and poorly disperses in the resin, fails to impart luster on the surfaces of the molded articles of a resin blended therewith, and offers poor appearance and decreased

Further, the known zinc borate has been synthesized
in an aqueous medium containing sodium salts and, hence,
contains sodium components as impurities in considerably
large amounts. When the plastic molded articles for
electric and electronic parts are blended with such a zinc
borate for a purpose of imparting flame-retarding
property, it is probable that the electric properties are
deteriorated as represented by poor insulation and
dielectric breakdown after the use for extended periods of
time being affected by humidity.
(Disclosure of the Invention)

It is, therefore, an object of the present invention to provide a zinc borate having a particular crystallite size and containing very little sodium components, and a method of preparing the same.

Another object of the present invention is to provide
a zinc borate which excellently disperses in a resin,
markedly improves smoothness, luster and appearance of the
surfaces of a resin blended therewith, and maintains, on
an excellent level, the electric properties of the resin
blended therewith as a flame-retarding agent or a smokesuppressing agent, and a method of preparing the same.

According to the present invention, there is provided a zinc borate having a chemical composition represented by the following formula (1).

 $2ZnO \cdot mB_2O_3 \cdot XH2O$ --- (1)

5 wherein m is a number of from 2.8 to 3.2, and x is a number of not larger than 4, and having a crystallite size of not smaller than 40 nm as found from diffraction peaks of indexes of planes of (020), (101) and (200) in the X-ray diffraction (Cu-kα) 10 and containing sodium components in amounts of not larger than 100 ppm as measured by the atomic absorptiometric method.

In the zinc borate according to the present invention, it is desired that the individual particles are independent rhombic hexahedrons, the length of a side of each particle lying in a range of from 0.3 to 7.0 μ m as measured by a scanning-type electron microphotograph.

In the zinc borate according to the present invention, it is desired that a product of crystallite sizes as found from the diffraction peaks of indexes of planes (020), (101) and (200) is not smaller than 200,000 nm³, and that a volume-based median diameter as found by a laser diffraction method is in a range of from 1.0 to 6.0 pm.

The invention further provides a method of preparing a zinc borate by forming fine crystals of a zinc borate by reacting a zinc flower and a boric acid at a substantially stoichiometric ratio at a relatively low temperature, effecting the aging as required and, then, maintaining the reaction system at a relatively high temperature to grow the crystals.

According to the present invention, further, there are provided a flame-retarding agent or a flame-retarding assistant, a smoke-suppressing agent, an antibacterial agent and a water glass-curing agent comprising the above

zinc borate.

(Brief Description of Drawings)

Fig. 1 shows an X-ray diffraction image (Cu-k α) of a zinc borate according to an embodiment 1 of the present 5 invention:

- Fig. 2 is a scanning-type electron microphotograph (magnification: 5000 times) showing the particle structure of the zinc borate according to the embodiment 1 of the present invention;
- Fig. 3 is a scanning-type electron microphotograph (magnification: 7000 times) showing, on an enlarged scale, the particle structure of the zinc borate according to the present invention;
- Fig. 4 is an X-ray diffraction image (Cu-k α) of a 15 zinc borate according to an embodiment 2 of the present invention:
- Fig. 5 is a scanning-type electron microphotograph (magnification: 5000 times) showing the particle structure of the zinc borate according to the embodiment 2 of the 20 present invention;
 - Fig. 6 shows an X-ray diffraction image (Cu-k α) of a zinc borate according to an embodiment 3 of the present invention:
- Fig. 7 is a scanning-type electron microphotograph
 25 (magnification: 5000 times) showing the particle structure
 of the zinc borate according to the embodiment 3 of the
 present invention;
- Fig. 8 shows an X-ray diffraction image (Cu-klpha) of a zinc borate according to an embodiment 4 of the present 30 invention;
 - Fig. 9 is a scanning-type electron microphotograph (magnification: 5000 times) showing the particle structure of the zinc borate according to the embodiment 4 of the present invention;
- 35 Fig. 10 shows an X-ray diffraction image (Cu-k α) of

a zinc borate according to an embodiment 5 of the present invention:

Fig. 11 is a scanning-type electron microphotograph
 (magnification: 5000 times) showing the particle structure
5 of the zinc borate according to the embodiment 5 of the
 present invention;

Fig. 12 shows an X-ray diffraction image $(Cu-k\alpha)$ of a zinc borate according to an embodiment 6 of the present invention:

10 Fig. 13 is a scanning-type electron microphotograph (magnification: 5000 times) showing the particle structure of the zinc borate according to the embodiment 6 of the present invention;

Fig. 14 shows an X-ray diffraction image $(Cu-k\alpha)$ of a zinc borate according to an embodiment 7 of the present invention;

Fig. 15 is a scanning-type electron microphotograph (magnification: 5000 times) showing the particle structure of the zinc borate according to the embodiment 7 of the 20 present invention;

Fig. 16 shows an X-ray diffraction image (Cu-k α) of a zinc borate according to an embodiment 8 of the present invention;

Fig. 17 is a scanning-type electron microphotograph
25 (magnification: 5000 times) showing the particle structure
of the zinc borate according to the embodiment 8 of the
present invention;

Fig. 18 shows an X-ray diffraction image (Cu-k α) of a zinc borate which is a comparative sample H-1;

Fig. 19 is a scanning-type electron microphotograph (magnification: 5000 times) showing the particle structure of the zinc borate which is the comparative sample H-1;

Fig. 20 shows an X-ray diffraction image (Cu-k α) of a zinc borate which is a comparative sample H-2;

35 Fig. 21 is a scanning-type electron microphotograph

(magnification: 5000 times) showing the particle structure of the zinc borate which is the comparative sample H-2;

Fig. 22 shows an X-ray diffraction image $(Cu-k\alpha)$ of a commercially available zinc borate (comparative sample H-3) manufactured by company A;

Fig. 23 is a scanning-type electron microphotograph (magnification: 5000 times) showing the particle structure of the commercially available zinc borate (comparative example H-3) manufactured by company A; and

Fig. 24 shows an X-ray diffraction image (Cu-kα) of a commercially available zinc borate (comparative sample H-4) manufactured by company B.

Fig. 25 shows a peak found by decreasing the scanning speed of an index of a plane (020) of Fig. 1, as a 15 representative example for finding a half-value width of a diffraction peak.

(Best Mode for Carrying Out the Invention) [Action]

The zinc borate according to the present invention 20 has a chemical composition represented by the following formula (1),

 $2ZnO \cdot mB_2O_3 \cdot XH_2O$ --- (1)

wherein m is a number of from 2.8 to 3.2, and x is a number of not larger than 4.

25 A variety kinds of zinc borates have been known, such as,

 $ZnO \cdot B_2O_3 \cdot 1 \sim 2H_2O$, $2ZnO \cdot 3B_2O_3 \cdot 3 \sim 9H_2O$, $3ZnO \cdot 5B_2O_3 \cdot 14H_2O$, $ZnO \cdot 5B_2O_3 \cdot 4 \cdot 5H_2O$, $6ZnO \cdot 5B_2O_3 \cdot 3H_2O$.

Among them, however, the present invention deals with the zinc borates of the 2.3 type having the most excellent flame-retarding property (hereinafter those of the 2.3 35 type are simply referred to as the zinc borates).

The zinc borates according to the present invention exhibit the same X-ray diffraction image as that of the conventional zinc borate of the 2.3 type.

Fig. 1 of an accompanying drawing shows an X-ray

5 diffraction image ($Cu-k\alpha$) of the zinc borate according to the present invention, and the following Table 1 shows relationships among the spacings, indexes of planes and relative intensities of the diffraction image.

10 <u>Table 1</u>

	2θ	Spacing (A)	Index of a plane	Relative Intensity
15	18.0	4.91	(020)	100
	20.6	4.31	(101)	78.2
	21.7	4.08	(120)	75.1
	22.5	3.95	(111)	21.7
	23.7	3.75	(121)	74.7
20	24.1	3.69	(200)	40.0
	27.5	3.32	(121)	22.0
	28.7	3.11	(012)	63.3

In the X-ray diffraction of crystals, it has been 25 known that an intensity peak appears in the interference when the following Bragg's formula (2),

$$n\lambda = 2dhkl \sin \theta$$
 --- (2)

wherein n is a degree, λ is a wavelength of an X-ray, dhkl is a spacing of (hkl) of a crystal , and θ is an angle of diffraction,

is satisfied, and there also exists a relationship represented by the following Scherrer's formula (3) between the sharpness of the interference peak and the size of the crystal,

35 Lhkl =
$$K\lambda/(H\cos\theta)$$
 --- (3)

wherein Lhkl is a size of a crystal in a direction perpendicular to a plane (hkl). K is a constant and is about 0.9, H is a half-value width (radian) of an interference peak, and λ and θ are the same as those of the above-mentioned formula (2).

The zinc borate according to the present invention has a feature in that the crystallite sizes found from the diffraction peaks of the indexes of planes (020), (101) and (200) in the X-ray diffraction (Cu-ka) are all not 10 smaller than 40 nm, and that the crystallite sizes are expanded as compared with those of the conventional zinc borates.

The zinc borate of the present invention is compared below in Table 2 with a commercially available zinc borate 15 manufactured by company A concerning their indexes of planes, half-value widths and crystallite sizes.

Table 2

20	Bragg angle	Index of	This	Invention	Produc	t of A Co.
	<u>(2 \theta)</u>	plane	Half- value width	Crystal- lite size	Half- value <u>width</u>	Crystal- lite size
25						
	18.03	020	0.119	67.6	0.146	55.1
	20.54	101	0.126	64.1	0.161	50.1
	24.10	200	0.126	64.5	0.159	51.1

30 The zinc borate according to the present invention has an additional feature in that a product of the crystallite sizes as found from the diffraction peaks of indexes of planes (020), (101) and (200) is not smaller than 200,000 nm3 and, particularly, not smaller than

250,000 nm3. 35

Concerning the zinc borates shown in Table 2, the Concerning the zinc borates shown in Table 21 the in Concerning the zinc borates shown to be 141,062 nm in Concerning the zinc borates found to be 141,062 nm in common to be 141,062 n product of crystallite sizes is found to be 141,062 mm in the company A, product of crystallite sizes manufactured by the company A, the case of the zinc borete manufactured by horse of the zinc borete manufactured by horse of the zinc bore case of the case of the zinc bore case of the the case of the sinc borste manufactured by the company A the case of the sinc borste case of the nowine that the case of the sinc borste case of the nowine that the case of the since the since the case of the since the and 15 279,489 mm in the case of the zinc borate the fine from which it is download that the from which it is one in horate the from which is one in horat the from which is one in horat the from which is one in horat the first of the first one is one in his one in his one in his one in his one is one in his one in his one in his one in his one is one in his one in his one in his one in his one is one in his one in ne of the crystallite is nearly doubled.

It is believed that account on the manner of the crystallite is nearly of the crystallite present invention from which it is obvious to It is believed that the size of the crystallite is giving a very favorable effect on the structure of zinc giving a very favorable affect on the succession of a continuous cont te Particles of definite shapes in has a feature in the since of the invention has a feature in the since in The lind wind a length of a side of each next in the the them is a feature in the lind with the lind of a side of each next in the them. that the individual particles are independent thombic in the individual particles are independent article in a canning the the individual particles are independent for a canning the the individual particles are independent by a canning that the individual particles are independent by a canning that the individual particles are independent individual particles in a canning the individual particles are independent thombic. giving a very ravorable errect on the giving a very ravorable errect of the giving a very ravorable errect on the giving a very ravorable errect o heraledrone having a length of a side of each particle in the state of the second by a scanning a range of trom 0.3 to 7.0 µm as measured by a scanning a range of trom mirronnovarrann. electron microphotograph. 1719. 238 a scanning.

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In the particle dismeter.

Uniformity in the particle

It is obvious that the zinc borate according to the present invention belongs to a so-called polycrystalline one since the crystallite size is greatly different from the real particle size. It should, however, be noted that the crystalline zinc borate of the present invention features a very high regularity in the arrangement of zinc borate crystallites.

Fig. 3 which is an attached drawing is a scanningtype electron microphotograph (magnification: 7000 times) 10 illustrating, on an enlarged scale, the structure of crystals of the zinc borate according to the present invention.

As will obvious from the above X-ray diffraction image, the zinc borate crystals belong to the monoclinic system. The zinc borate particles shown in Fig. 3 obviously has the structure of a rhombic hexahedron which is that of the monoclinic system, which is quite the same as a single crystal that cannot be distinguished, in appearance, from an aggregate of crystals.

Owing to the above crystal structure and particle

structure, the zinc borate of the present invention offers the following advantages. That is, the conventional zinc borate poorly disperses in the resin, fails to impart luster on the surfaces of the molded articles of a resin blended therewith, and offers poor appearance and decreased commercial value. Owing to the above crystal structure and particle structure, however, the zinc borate of the present invention excellently disperses in the resin, imparts smoothness and luster onto the surfaces of the molded articles of the resin blended therewith and, further, strikingly improves the appearance.

Being associated with the above crystal structure and particle structure, the zinc borate of the present invention further gives excellent advantages such as

35 imparting flame-retarding property and smoke-suppressing

property inherent in the zinc borate.

Being also associated with the method of its preparation, the zinc borate of the present invention has such a feature that the content of sodium component is not larger than 100 ppm, preferably, not larger than 50 ppm and, more preferably, not larger than 30 ppm as measured by the atomic absorptiometric method.

For instance, the above-mentioned zinc borate manufactured by the company A has a sodium content of 350 ppm, whereas the zinc borate of a particle structure shown in Fig. 3 has a sodium content of 15 ppm, which is a reduction to 1/20 or less of sodium content.

Deterioration in the electric properties of a resin composition blended with a zinc borate can be evaluated by 15 a promotion testing being immersed in hot water. In the promotion testing in Example appearing later, for example, a resin composition blended with 10 parts of the zinc borate manufactured by the company A exhibits a volume resistivity (80°C) of 4.06 x 10¹³ whereas the resin 20 composition blended with 10 parts by weight of the zinc borate of the invention exhibits a volume resistivity (80°C) of 8.02 x 10¹³, thus obviously imparting flameretarding property and smoke-suppressing property while maintaining electric properties of the resin composition blended with the zinc borate on an excellent level.

In preparing the zinc borate according to the present invention, a zinc flower and a boric acid are reacted together at a substantially stoichiometric ratio and at a relatively low temperature such as from 45 to 65°C to form 30 fine crystals of zinc borate, followed, as required, by aging to grow the crystals while maintaining the reaction system at a relatively high temperature, such as 70 to 100°C.

The method of the present invention selects a zinc 35 flower and a boric acid as starting materials for

synthesis in order to avoid the infiltration of electric property-deteriorating components such as sodium as much as possible. In order to synthesize the zinc borate having the above-mentioned crystal structure and particle 5 structure from the above starting materials, it is also important that these two starting materials are reacted together at a substantially stoichiometric ratio.

In the present invention, it is important that the reaction is conducted in two steps. First, the two are 10 reacted together at a low temperature to prepare fine crystalline zinc borate. In conducting the synthesis, formation of fine crystalline zinc borate can be easily confirmed by an increase in the viscosity of the reaction system.

15 Then, the formed fine crystals are aged by maintaining, as required, the reaction system at the reaction temperature in the first step or at a temperature slightly higher than the above temperature and, then, the crystals are grown by maintaining the reaction system at a temperature higher than the reaction temperature in the first step. Further, the reaction in the first step may be conducted by adding seed crystals of zinc borate in an amount of from 0.5 to 10 parts by weight per 100 parts by weight of the B₂O₃ component of boric acid, in order to 25 shorten the reaction time.

Concretely speaking, when there exists no seed crystal, it is desired to conduct the reaction in the first step at a temperature of from 40 to 75°C and, preferably, from 45 to 70°C and to conduct the reaction in 30 the second step at a temperature of from 70 to 120°C and, preferably, from 75 to 110°C. When there exist seed crystals, it is desired to conduct the reaction in the first step at a temperature of from 45 to 70°C and, preferably, from 50 to 70°C and to conduct the reaction in the second step at a temperature of from 75 to 120°C and,

preferably, from 75 to 110°C.

According to the present invention which conducts the reaction in two steps as described above, it is allowed to prepare the zinc borate having the above-mentioned crystal 5 structure and particle structure.

The zinc borate according to the present invention can be effectively used as a flame-retarding agent or a flame-retarding assistant, a smoke-suppressing agent, an antibacterial agent, a water glass-curing agent and the

Among these uses, the zinc borate of the invention is effectively used being blended in the resin to express properties of the above-mentioned agents.

As the resin to be blended with, there can be

15 exemplified any thermoplastic resin, elastomer,
thermosetting resin or blends thereof. The zinc borate
according to the present invention is used in an amount of
from 1 to 150 parts by weight and, particularly, from 3 to
30 parts by weight per 100 parts by weight of the resin.

20 The zinc borate exhibits excellent flame-retarding effect
since ① the dehydrating endothermic reaction due to
thermal decomposition lowers the temperature of
combustion, and ② zinc works as a catalyst for the
dehalogenation reaction, promotes the formation of a

25 carbonized layer and suppresses the generation of smoke. As the thermoplastic resin, there can be used a resin synthesized by using a metallocene catalyst, as well as polyolefins such as low-density polyethylene, high-density polyethylene, polypropylene, poly-1-butene, poly-4-methyl-30 1-pentene and α-olefin random or block copolymers like

ethylene, propylene, 1-butene and 4-methyl-1-pentene; styrene resins such as ethylene/vinyl acetate copolymer, ethylene/vinyl alcohol copolymer, polystyrene,

acrylonitrile/styrene copolymer, ABS, and α -methyl styrene/styrene copolymer; polyvinyl compounds such as

methyl acrylate and methyl polymethacrylate; polyamides such as nylon 6, nylon 6-6, nylon 6-10, nylon 11 and nylon 12; thermoplastic polyesters such as polyethylene terephthalate and polybutylene terephthalate;

5 polycarbonate; polyphenylene oxide; or mixtures thereof. In particular, the zinc borate of the present invention exhibits a great effect when it is used being blended in a chlorine-containing polymer. As the chlorine-containing polymer, there can be exemplified polymers such as

10 polyvinyl chloride, polyvinylidene chloride, chlorinated polyethylene, chlorinated polypropylene, chlorinated rubber, vinyl chloride/vinyl acetate copolymer, vinyl chloride/ethylene copolymer, vinyl chloride/propylene copolymer, vinyl chloride/styrene copolymer, vinyl

15 chloride/isobutylene copolymer, vinyl chloride/vinylidene chloride copolymer, vinyl chloride/styrene/acrylonitrile copolymer, vinyl chloride/butadiene copolymer, vinyl chloride/propylene chloride copolymer, vinyl chloride/vinylidene chloride/vinyl acetate tercopolymer,

20 vinyl chloride/styrene/maleic anhydride tercopolymer, vinyl chloride/acrylic acid ester copolymer, vinyl chloride/maleic acid ester copolymer, vinyl chloride/methacrylic acid ester copolymer,

vinylchloride/acrylonitrile copolymer and internally
25 plasticized polyvinyl chloride; as well as blends of these
chlorine-contained polymers with polyethylene, polybutene,
ethylene/vinyl acetate copolymer, ethylene/propylene
copolymer, polystyrene, acrylic resin,

acrylonitrile/butadiene/styrene copolymer, or acrylic acid 30 ester/butadiene/styrene copolymer.

As the elastomer, there can be exemplified nitrile/butadiene rubber (NBR), styrene/butadiene rubber (SBR), chloroprene rubber (CR), polybutadiene (BR), polyisoprene (IIB), butyl rubber, natural rubber, ethylene/propylene rubber (EPR), ethylene/propylene/diene

rubber (EPDM), polyurethane, silicone rubber and acrylic rubber; and thermoplastic elastomers such as styrene/butadiene/styrene block copolymer, styrene/isoprene/styrene block copolymer, hydrogenated 5 styrene/butadiene/styrene block copolymer, and hydrogenated styrene/isoprene/styrene block copolymer.

As the thermosetting resin, there can be exemplified phenol-formaldehyde resin, furan-formaldehyde resin, xylene-formaldehyde resin, ketone-formaldehyde resin, 10 urea-formaldehyde resin, melamine-formaldehyde resin, alkyd resin, unsaturated polyester resin, epoxy resin, bismaleimide resin, triallylcyanurate resin, thermosetting acrylic resin, silicone resin and urethane resin. These resins can be used alone or in a combination of two or 15 more kinds. In particular, the epoxy resin generally refers to monomers, oligomers and polymers having two or more epoxy groups in one molecule thereof, but is not to limit their molecular weights or molecular structures. Its examples include biphenyl epoxy compound, bisphenol 20 epoxy compound, phenol novolak epoxy resin, cresol novolak resin, triphenolmethane epoxy compound and alkyl-modified triphenolmethane epoxy compound, which may be used alone or being mixed together.

The zinc borate of the invention has a feature in that the component of sodium component is not larger than 100 ppm, preferably, not larger than 50 ppm and, more preferably, not larger than 30 ppm as measured by the atomic absorptiometric method, and is best suited for an epoxy resin for sealing semiconductor. When an epoxy resin is used, the curing agent is, preferably, phenol novolak resin, dicyclopentadiene-modified phenol resin, paraxylylene-modified phenol resin or terpene-modified phenol resin, and the curing-promoting agent is, preferably, 1,8-diazabicycloundecene, triphenylphosphine, benzyldimethylamine or 2-methylimidazole.

The zinc borate of the present invention can be used being blended in a resin, the zinc borate being used in a single kind as a flame-retarding component or being used in combination with one or more of other flame-retarding 5 agents such as aluminum hydroxide, magnesium hydroxide, hydrotalcite compound, zinc-modified hydrotalcite-like compound, lithium aluminum composite hydroxide salt, polybasic aluminum magnesium salt, zinc-modified polybasic aluminum magnesium salt, dawsonite, polyhydric alcohol, 10 polyhydric alcohol partial ester and epoxy compound. Those having hardnesses smaller than that of the zinc borate may be ground with the flame-retarding agent, and may be applied to the surfaces of the zinc borate of the invention. In this case, the amount of application is 15 from 0.5 to 50 parts by weight and, preferably, from 0.5 to 30 parts by weight per 100 parts by weight of the zinc

For example, the mixing under the grinding condition by using an alkaline earth metal compound stands for a 20 mixing in which an alkaline earth metal compound is ground with zinc borate particles, and the zinc borate particles are smeared with fine particles of the alkaline earth metal compound formed by the grinding. In the grindmixing, the zinc borate particles are very harder than the 25 alkaline earth metal compound and, hence, work as a pulverizing medium for the alkaline earth metal compound.

Henschel's mixer, super mixer, tube mill, ball mill, vibration mill, pin mill, mixing and grinding machine or atomizer can be used for the grind-mixing.

30 [Hydrotalcite]

borate.

Hydrotalcite is a synthetic mineral that belongs to an aluminum magnesium carbonate hydroxide, and there is used a composite metal hydroxide having the following general formula (4),

35
$$M2_xM3_y(OH)_{2x + 3y - 2z} (A^{2-})_z \cdot AH2O$$
 --- (4

30

wherein M2 is a divalent metal ion such as Mg, M3 is a trivalent metal ion such as Al, A2- is a divalent anion such as CO3, and x, y, z are positive numbers satisfying $8 \ge x/y \ge 1/4$ and z/x+y > 1/20, and a is a number satisfying $0.25 \le a/x+y \le 1.0$.

Among these composite metal hydroxides, a compound represented by the following formula (5),

Mq6Al2(OH)16(CO3) · 4H2O --- (5)

is a natural mineral known as hydrotalcite. This mineral 10 and homologues are synthesized by methods taught in Japanese Examined Patent Publication (Kokoku) Nos. 32198/1972, 29477/1973 and 29478/1973 filed by Kyowa Kagaku Co.

In particular, it has been known that a compound represented by the following formula (6), 15

 $Mq_{4.5}Al_2(OH)_{13}(CO_3) \cdot 3H_2O$ --- (6) exhibits excellent chlorine ion-trapping property.

It is also allowable to use a compound in which perhalogen oxyacid ions are introduced by utilizing a 20 property that the hydrotalcites are easily ion-exchanged in a state in which they have been dispersed well in water, i.e., by utilizing a property that the carbonic acid ions are exchanged with other anions.

Concretely, the hydrotalcite compounds have 25 composition formulas Mq5Al2(OH)16CO3.4H2O, $Mq_{4.5}Al_{2}(OH)_{13}CO_{3} \cdot 3.5H_{2}O$, $Mq_{0.66}Al_{0.34}(OH)_{2}(SiO_{3})_{0.17} \cdot 0.52H_{2}O$, $Mg_{0.7}Al_{0.3}(OH)_2(CO_3)_{0.15} \cdot 0.55H_2O$, etc. They have such trade names as Alkamizer 1, Alkamizer 2, Alkamizer 3, Alkamizer 4, DHT-4A, Kyoword, etc.

As the polyhydric alcohol that constitutes a polyhydric alcohol and polyhydric alcohol partial ester, there can be exemplified ethylene glycol, diethylene glycol, triethylene glycol, polyethylene glycol, trimethylene glycol, tetramethylene glycol, hexamethylene

35 glycol, neopentyl glycol, glycerin, diglycerin,

dipentaerythritol, manitol, sorbitol, trimethylolpropane, ditrimethylolpropane, trisisocyanurate, monopentaerythritol, and dipentaerythritol adipate.

Preferably, there is used a monopentaerythritol having an average particle diameter of from 0.1 to 100 µm.

Further, the above polyhydric alcohols and esters thereof may be used alone or in a combination of at least one or more kinds selected out of them.

In addition to the above, there can be used oxides,

10 hydroxides or sulfates of antimony, zirconium and

molybdenum, zinc stannate, zinc hydroxystannate, halogencontaining flame-retarding agent, phosphoric acid estertype flame-retarding agent and halogenophosphoric acid
ester-type flame-retarding agent in one kind or in a

15 combination of two or more kinds.

As the antimony-type flame-retarding agent, there can be favorably used antimony trioxide, antimony pentoxide and sodium antimonate. There can be further used trimethyl stilbene, triethyl stilbene and triphenyl 20 stilbene.

As the zinc stannate-type or zinc hydroxystannatetype flame-retarding agent, there is used a composition represented by the formula (7),

 $ZnSnO_3$ or $ZnSn(OH)_6$ --- (7)

As the halogen-type flame-retarding agent, there can be exemplified aliphatic halide compounds such as 1,2dichloroethane, 1,2-dibromoethane, 1,1,2,2tetrachloroethane, 1,1,2,2-tetrabromoethane, hexachloroethane, hexabromoethane,

- 30 dibromotetrachloroethane, 1,2,3,4-tetrachlorobutane, 1,2,3,4-tetrabromobutane, chlorinated paraffin and brominated paraffin; aliphatic halogen compounds such as pentabromomonochlorocyclohexane, hexabromocyclohexane, hexachlorocyclohexane, hexabromocyclodecane,
- 35 hexachlorocyclodecane, hexachlorocyclopentadiene,

hexabromocyclopentadiene, chloroendoic acid, diallyl chloroendoate, anhydrous chloroendoic acid and similar iodine compounds; and aromatic halogen compounds such as hexabromobenzene, hexachlorobenzene,

- 5 pentabromomethylbenzene, pentachloromethylbenzene, hexabromodiphenyl, hexachlorodiphenyl, hexabromodiphenyl ether, hexachlorodiphenyl ether, dibromocredylglycidyl ether, decabromobiphenyl ether, decachlorobiphenyl ether, decabromodiphenyl oxide, decachlorodiphenyl oxide,
- 10 octabromodiphenyl ether, octachlorodiphenyl ether, tribromophenol, trichlorophenol, tetrabromobisphenol A, tetrachlorobisphenol A, tetrabromobisphenol F, tetrabromobisphenol AD, dibromodichlorobisphenol A, diacetate of tetrabromobisphenol A, diacetate of
- tetrachlorobisphenol A, tetrabromo-2,2-bis(4,4'dimethoxyphenyl)propane, tetrachloro-2,2-bis(4,4'dimethoxyphenyl)propane, tetrabromophthalic anhydride,
 tetrachlorophthalic anhydride, chlorinated epoxy novolak
 phenol resin, brominated epoxy novolak phenol resin,
- 20 brominated bisphenol A epoxy resin and similar iodine compounds.

As the phosphoric acid ester-type flame-retarding agent, there can be exemplified trimethyl phosphate, triethyl phosphate, triottyl phosphate, triottyl

25 phosphate, tributoxyethyl phosphate, octyldiphenyl phosphate, tricresyl phosphate, triphenyl phosphate and cresyldiphenyl phosphate.

As the halogenophosphoric acid ester-type flameretarding agent, there can be exemplified
30 tris(chloroethyl)phosphate, tris(2-chloropropyl)phosphate,
tris(2,3-dichloropropyl)phosphate, tris(2,3dibromopropyl)phosphate, and
tris(bromochloropropyl)phosphate.

From the standpoint of suppressing the generation of 35 smoke and imparting flame-retarding property, it is desired to use these frame-retarding agents at a weight ratio of from 1:20 to 20:1 and, particularly, from 1:10 to 10:1 relative to the zinc borate. Even in case fire has broken, the flame-retarding property and smoke-suppressing property facilitate evacuation, rescue operation and fire-extinguishing activity.

When a chlorine-contained polymer is used as a resin, it is desired that the resin is blended with a smoke-suppressing agent together with known blending agents such as plasticizer, lubricant, main heat stabilizer, assistant stabilizer, coloring agent, aging stabilizer, aging-retarding agent, photo-stabilizer, ultraviolet rayabsorbing agent, antistatic agent, reinforcing agent, reforming resin or rubber, basic inorganic metal salt, chelate agent, antioxidant, epoxy compound and other reinforcing agents or fillers.

As the plasticizer, there can be used the ones that have been known as a plasticizer for chlorine-contained polymers, such as phthalic acid ester, trimellitic acid ester, pyromellitic acid ester, aliphatic dibasic acid ester, phosphoric acid ester, hydroxypolyhydric carboxylic acid ester, monoaliphatic acid ester, polyhydric alcohol ester, epoxy-type plasticizer and polyester-type plasticizer.

25 As the lubricant, there can be used a variety of waxes such as petroleum wax, polyethylene wax, polypropylene wax, fatty acid or derivatives thereof, and plant waxes.

The amounts of blending the plasticizer and the

lubricant vary depending upon the use of the chlorinecontained polymer, i.e., depending upon the soft blending
or the hard blending. In the former case, the plasticizer
is blended in an amount of from 20 to 100 parts by weight
and, particularly, from 30 to 80 parts by weight and the

lubricant is blended in an amount of from 0.05 to 5 parts

by weight and, particularly, from 0.5 to 3 parts by weight per 100 parts by weight of the resin. In the latter case, the plasticizer is blended in an amount of from 0 to 10 parts by weight and, particularly, from 0 to 5 parts by weight and the lubricant is blended in an amount of from 0.1 to 5 parts by weight and, particularly, from 0.2 to 3 parts by weight per 100 parts by weight of the resin.

As the main stabilizer, there can be used known ones, such as lead-type stabilizers and non-lead-type

10 stabilizers in a single kind or in two or more kinds in combination.

As the lead-type stabilizer, there can be used any known ones and, particularly, tribasic to tetrabasic lead sulfate, basic lead phosphite, basic lead silicate, basic lead carbonate, basic lead maleate, basic lead phthalate, basic lead stearate, and higher fatty acid lead, which may be used in two or more kinds in combination.

As the non-lead-type stabilizer, there can be exemplified inorganic non-lead-type stabilizer, metal 20 soap-type stabilizer and organotin-type stabilizer. As the inorganic non-lead-type stabilizer, there can be used alkaline earth metal silicate-type stabilizer, alkaline earth metal aluminosilicate-type stabilizer, and alkaline earth metal or zinc-aluminum composite hydroxide 25 carbonate.

Preferred examples of the alkaline earth metal silicate-type stabilizer include a fine crystalline calcium silicate having a chemical composition represented by the general formula (8),

$$CaO \cdot xSiO_2 \cdot NH_2O$$
 --- (8)

wherein x is a number of not smaller than 0.5, and n is a number of not larger than 2.5, and having X-ray diffraction images in a spacing of from 3.01 to 3.08 angstroms, in a spacing of from 2.78 to 2.82 angstroms and in a spacing of from 1.81 to 1.84 angstroms,

or a composite product thereof with a polyhydric alcohol. Preferred examples of the polyhydric alcohol include pentaerythritol and dipentaerythritol.

Suitable examples of the alkaline earth metal
5 aluminosilicate-type stabilizer include those of the Atype, X-type, Y-type, L-type, P-type, T-type (nepheline),
as well as those of various crystal structures, such as
offretite, erionite, mordenite, ferrierite,
clinoptirolite, chabazite, analcime and aluminosilicate of
10 the sodalite group. However, A-type zeolite is

particularly preferred from the standpoint of hydrogen chloride-trapping property.

As the metal soap-type stabilizer, there are used calcium stearate, magnesium stearate, barium stearate and 15 zinc stearate in a single kind or in a combination of two or more kinds.

As the organotin-type stabilizer, there are used dibutyltin dilaurate, dibutyltin maleate, organotin mercaptide, di-n-octyltin laurate, di-n-octyltin maleate polymer, di-n-octyltinbis-2-ethylhexyl maleate, and di-n-octyltinbisisooctylthio glycolate.

It is desired that the above stabilizers are used in an amount of from 0.1 to 20 parts by weight and, particularly, from 0.5 to 10 parts by weight per 100 parts by weight of the resin from the standpoint of heat stability and preventing the initial coloring.

The stabilizers may be used in a single kind or in a combination of two or more kinds. From the standpoint of not decreasing the oxygen index concentration of the

30 chlorine-contained polymer, however, it is desired to use chiefly an inorganic stabilizer.

It is desired that the chlorine-contained polymer composition is further blended with β -diketone or β -keto acid ester in an amount of from 0.05 to 10 parts by weight 35 and, particularly, from 0.1 to 3 parts by weight per 100

parts by weight of the resin. Use the above chelating agent component effectively prevents the initial coloring.

As the β-diketone or β-ketoacid ester, there can be used, for example, 1,3-cyclohexadion, methylenebis-1,3-cyclohexadion, 2-benzyl-1,3-cyclohexadion, acetyltetralon, palmitoyltetralon, stearcyltetralon, benzoyltetralon, 2-acetylcyclohexanone, 2-benzoylcyclohexanone, 2-acetyl-1,3-cyclohexanedion, bis(benzoyl)methane, benzoyl-p-chlorobenzoylmethane, bis(4-methylbenzoyl)methane, bis(2-hydroxybenzoyl)methane, benzoylacetone, tribenzoylmethane, diacetylbenzoylmethane, stearcylbenzoylmethane, palmtoylbenzoylmethane, lauroylbenzoylmethane, dibenzoylmethane, bis(4-chlorobenzoyl)methane, bis(methylene-3,4-dioxybenzoyl)methane, benzoylacetylphenylmethane, stearcyl(4-

methoxybenzoyl)methane, butanoylacetone, distearoylmethane, acetylacetone, stearoylacetone, bis(cyclohexanoyl)methane and dipivaloylmethane.

chain reaction.

It is further desired that the chlorine-contained
polymer composition and the polyolefin-type resin contain,
in addition to the above components, a phenol-type
antioxidant in an amount of from 0.005 to 3 parts by
weight and, particularly, from 0.01 to 0.5 parts by weight
per 100 parts by weight of the resin component. The
antioxidant is effective in improving the heat stability
and in suppressing the heat degradation caused by the

As the phenol-type antioxidant, there can be used either a bisphenol-type antioxidant or a steric hindrance 30 phenol-type antioxidant. Examples include bisphenol A, bisphenol B, bisphenol F, 2,6-diphenyl-4-octadecyloxyphenol, stearyl(3,5-di-tertiary butyl-4-hydroxyphenyl)propionate, distearyl(3,5-di-tertiary butyl-4-hydroxybenzyl)phosphonate, 1,6-hexamethylenebis[(3,5-di-tertiary butyl-4-hydroxyphenyl)propionate], 1,6-

hexamethylenebis[(3,5-di-tertiary butyl-4-hydroxyphenyl)amide propionate], bis[3,3-bis(4-hydroxy-3-tertiary butylphenyl)butylic acid]glycol ester, 1,1,3-tris(2-methyl-4-hydroxy-5-tertiary butylphenyl)butane,

- 5 1,3,5-tris(2,6-dimethyl-3-hydroxy-4-tertiary butylbenzyl)isocyanurate, 1,3,5-tris(3,5-di-tertiary butyl-4-hydroxybenzyl)isocyanurate, and triethylene glycolbis[(3-tertiary butyl-4-hydroxy-5methylphenyl)propionate].
- As the ultraviolet ray-absorbing agent, there can be exemplified 2-hydroxybenzophenones such as 2,4-dihydroxybenzophenone, 2-hydroxy-4-methoxybenzophenone, and 5,5'-methylenebis(2-hydroxy-4-methoxybenzophenone); and 2-(2'-hydroxyphenyl)benzotriazoles such as 2-(2'-hydroxy-5'-methylphenyl)benzotriazole, 2-(2'-hydroxy-3'-tertiary butyl-5'-methylphenyl)-5-chlorobenzotriazole, 2-(2'-hydroxy-5'-tertiary octylphenyl)benzotriazole, 2-(2'-hydroxy-3',5'-dicumylphenyl)benzotriazole, and 2,2'-methylenebis(4-tertiary octyl-6-benzotriazolyl)benol.
- As the photo stabilizer, there can be exemplified hindered amine photo stabilizers, such as 1,2,2,6,6-pentamethyl-4-piperidyl stearate, 2,2,6,6-tetramethyl-4-piperidyl benzoate, N-(2,2,6,6-tetramethyl-4-piperidyl)dodecylimide succinate, 1-[(3,5-di-tertiary butyl-4-hydroxyphenyl)propionyloxyethyl]-2,2,6,6-
- tetramethyl-4-piperidyl-(3,5-di-tertiary butyl-4-hydroxyphenyl)propionate, bis(2,2,6,6-tetramethyl-4-piperidyl)sebacate, tetra(2,2,6,6-tetramethyl-4-piperidyl)butanetetracarboxylate, tetra(1,2,2,6,6-
- 30 pentamethyl-4-piperidyl)butanetetracarboxylate, bis(2,2,6,6-tetramethyl-4-piperidyl)-di(tridecyl)butane tetracarboxylate and bis(1,2,2,6,6-penamethyl-4piperidyl)-di(tridecyl)butane tetracarboxylate.
- To blend a chlorine-contained polymer or the like 35 polymer with the above components to be blended, the

components to be blended except plasticizer and/or lubricant are blended in advance as a one-package blending agent which, as required is granulated, and is mixed into the chlorine-contained polymer together with the

5 plasticizer and/or the lubricant by using a kneader such as roll mixer, Bumbery's mixer or pelletizer to obtain a hard composition for molding pipes or a soft composition for covering the electric wires, or to obtain a plastisol, which is then molded into films, sheets, tiles or any 10 other molded articles by calender machining, meltextrusion molding or slash-molding, or is used for coating.

Further, the zinc borate of the present invention can be used as an antibacterial agent or a water glass-curing 15 agent. In the field of use as antibacterial agents, the zinc borate of the invention can be used as fiber products such as film or sheet for holding freshness (atmosphere sterilization, controlling bacteria), polyester, polyethylene, polypropylene, nylon and acrylic resin; 20 building materials such as antibacterial paper, corrugated

cardboard (eradicating bacteria, controlling bacteria, controlling fungi), wall materials, ceiling materials, carpeting materials, floor materials, mats and roof underlying materials; toiletries, cosmetics, paints (eradicating bacteria, controlling bacteria, controlling fungi), splash or spray type bacteria eradicator, goods for kitchen and bath room (bags, containers, cooking

for kitchen and bath room (bags, containers, cooking board, drainboard, etc.), fishing nets and algae-controlling agent. The zinc borate can be further used

30 being mixed in a cement mortar or a cement concrete to produce antibacterial cement mortar and cement concrete (inclusive of products produced on the site). The zinc borate can be further used for a variety of products for controlling bacteria.

35 The zinc borate of the present invention having a

particular crystallite size and containing very small amounts of sodium component, disperses excellently in a resin, markedly improves smoothness and luster on the surfaces of the resin blended therewith, and works to greatly improve the appearance, and further maintains, on an excellent level, the electric properties of the resin blended therewith as a flame-retarding agent or smoke-suppressing agent. In particular, the zinc borate of the present invention improves the flame-retarding property of the chlorine-contained resins, nylons and epoxy resins.

In the following Examples, measurements were taken in accordance with the methods described below.

(1) Average particle diameter and particle size.

Average particle diameters and average particle sizes were measured by using a particle size analyzer, Model LS230, manufactured by Coulter Co.

(2) X-Ray diffraction.

Measured with Cu-K α by using a Geigerflex RAD-B 20 system manufactured by Riqaku Denki Co.

Target: Cu

Filter: Curved crystalline graphite monochrometer Detector: SC

Voltage: 40 KV

25 Current: 20 mA

30

Full-scale count: 700 c/s

Smoothing point: 25

Scanning speed: 2°/min

Step sampling: 0.02°

Slit: DS 1° RS 0.15 mm SS 1°

Irradiation angle: 6°

(3) $X\sim Ray$ diffraction condition in measuring crystallite size.

Measured with $Cu-K\alpha$ by using a Geigerflex RAD-B system manufactured by Rigaku Denki Co.

Target: Cu Filter: Ni

Tube voltage: 40 KV

Tube current: 20 mA

5 Full-scale count: 4 kcps Scanning speed: 0.25°/min Time constant: 0.5 sec

Slit: DS(SS) 0.5° RS 0.15 mm

Irradiation angle: 6°

Fig. 25 shows a peak from which an index of a plane(020) of Fig. 1 is found, as a representative example for finding a hald-value width of a diffraction peak.

(4) SEM measurement.

Measured by using a scanning electron microscope, S-15 570, manufactured by Hitachi, Ltd.

(5) Measurement of atomic absorbency.

Measured by using z-8200 manufactured by Hitachi, Ltd.

(6) Chemical analysis

20 Conducted in compliance with the chemical analysis of lime stipulated under JIS R9011 (Example 1)

An aqueous solution was prepared by dissolving 72.5 g of boric acid (B₂O₃ content of 56.3%) in 1000 ml of pure 25 water. To the aqueous solution were added 96.0 g of a zinc flower (ZnO content of 99.4%) and 217.5 g of boric acid (B₂O₃ content of 56.3%), and were stirred and mixed together such that the molar ratio of B₂O₃/ZnO was 2.0. Next, the solution was stirred and reacted at 60°C for 90 minutes. The solution was further stirred and reacted at 90°C for 4 hours. The obtained product was filtered, washed with water and was, then, dried at 105°C to obtain a zinc borate (sample A-1). Table 3 shows the chemical composition and properties of the thus obtained zinc

35 borate (sample A-1), Fig. 1 shows an X-ray diffraction

image thereof, and Figs. 2 and 3 show electron microphotographs thereof.

(Example 2)

- An aqueous solution was prepared by dissolving 72.5 q 5 of boric acid (B2O3 content of 56.3%) in 1000 ml of pure water to which has been added 1,25 g of zinc borate $(2Zn0 \cdot 3B_2O_3 \cdot 3.5H_2O)$ as a seed in advance. To the aqueous solution were added 95.7 g of a zinc flower (ZnO content of 99.4%) and 217.5 g of boric acid (B2O3 content of 10 56.3%), and were stirred and mixed together such that the molar ratio of B2O3/ZnO was 2.0. Next, the solution was stirred and reacted at 60°C for 90 minutes. The solution was further stirred and reacted at 90°C for 4 hours. obtained product was filtered, washed with water and was, 15 then, dried at 105°C to obtain a zinc borate (sample A-2). Table 3 shows the chemical composition and properties of the thus obtained zinc borate (sample A-2), Fig. 4 shows an X-ray diffraction image thereof, and Fig. 5 shows an electron microphotograph thereof.
- 20 (Example 3)

An aqueous solution was prepared by dissolving 72.5 g of boric acid (B₂O₃ content of 56.3%) in 1000 ml of pure water. To the aqueous solution were added 95.7 g of a zinc flower (ZnO content of 99.4%) and 217.5 g of boric acid (B₂O₃ content of 56.3%), and were stirred and mixed together such that the molar ratio of B₂O₃/ZnO was 2.0. Next, the solution was stirred and reacted at 45°C for 120 minutes. The solution was further stirred and reacted at 90°C for 4 hours. The obtained product was filtered, 30 washed with water and was, then, dried at 105°C to obtain a zinc borate (sample A-3). Table 3 shows the chemical composition and properties of the thus obtained zinc borate (sample A-3), Fig. 6 shows an X-ray diffraction image thereof, and Fig. 7 shows an electron

(Example 4)

(Example 5)

An aqueous solution was prepared by dissolving 72.5 g of boric acid (B₂O₃ content of 56.3%) in 1000 ml of pure water. To the aqueous solution were added 95.7 g of a 5 zinc flower (ZnO content of 99.4%) and 217.5 g of boric acid (B₂O₃ content of 56.3%), and were stirred and mixed together such that the molar ratio of B₂O₃/ZnO was 2.0. Next, the solution was stirred and reacted at 60°C for 120 minutes. The solution was further stirred and reacted at 10 80°C for 8 hours. The obtained product was filtered, washed with water and was, then, dried at 105°C to obtain a zinc borate (sample A-4). Table 3 shows the chemical composition and properties of the thus obtained zinc borate (sample A-4), Fig. 8 shows an X-ray diffraction image thereof, and Fig. 9 shows an electron microphotograph thereof.

An aqueous solution was prepared by dissolving 72.5 g of boric acid (B2O3 content of 56.3%) in 1000 ml of pure 20 water. To the aqueous solution were added 95.7 g of a zinc flower (ZnO content of 99.4%) and 217.5 g of boric acid (B2O3 content of 56.3%), and were stirred and mixed together such that the molar ratio of B2O3/ZnO was 2.0. Next, the solution was stirred and reacted at 60°C for 120 25 minutes. The solution was further stirred and reacted at 85°C for 6 hours. The obtained product was filtered, washed with water and was, then, dried at 105°C to obtain a zinc borate (sample A-5). Table 3 shows the chemical composition and properties of the thus obtained zinc 30 borate (sample A-5), Fig. 10 shows an X-ray diffraction image thereof, and Fig. 11 shows an electron microphotograph thereof. (Example 6)

An aqueous solution was prepared by dissolving 72.5 g 35 of boric acid (B_2O_3 content of 56.3%) in 1000 ml of pure

water to which has been added 1.25 g of zinc borate $(2\text{ZnO} \cdot 3\text{B}_2\text{O}_3 \cdot 3.5\text{H}_2\text{O})$ as a seed in advance. To the aqueous solution were added 95.7 g of a zinc flower (ZnO content of 99.4%) and 217.5 g of boric acid (B₂O₂ content of

- 5 56.3%), and were stirred and mixed together such that the molar ratio of B_2O_3/ZnO was 2.0. Next, the solution was stirred and reacted at 65°C for 80 minutes. The solution was further stirred and reacted at 90°C for 4 hours. The obtained product was filtered, washed with water and was,
- 10 then, dried at 105°C to obtain a zinc borate (sample A-6). Table 3 shows the chemical composition and properties of the thus obtained zinc borate (sample A-6), Fig. 12 shows an X-ray diffraction image thereof, and Fig. 13 shows an electron microphotograph thereof.

15 (Example 7)

An aqueous solution was prepared by dissolving 72.5 g of boric acid (B₂O₃ content of 56.3%) in 1000 ml of pure water. To the aqueous solution were added 95.7 g of a zinc flower (ZnO content of 99.4%) and 217.5 g of boric 20 acid (B₂O₃ content of 56.3%), and were stirred and mixed together such that the molar ratio of B₂O₃/ZnO was 2.0. Next, the solution was stirred and reacted at 55°C for 120 minutes. The solution was further stirred and reacted at 75°C for 7 hours. The obtained product was filtered,

25 washed with water and was, then, dried at 105°C to obtain a zinc borate (sample A-7). Table 3 shows the chemical composition and properties of the thus obtained zinc borate (sample A-7), Fig. 14 shows an X-ray diffraction image thereof, and Fig. 15 shows an electron

30 microphotograph thereof.
(Example 8)

An aqueous solution was prepared by dissolving 72.5 g of boric acid (B_2O_3 content of 56.3%) in 1000 ml of pure water. To the aqueous solution were added 95.7 g of a zinc flower (ZnO content of 99.4%) and 217.5 g of boric

acid (B₂O₃ content of 56.3%), and were stirred and mixed together such that the molar ratio of B₂O₃/ZnO was 2.0. Next, the solution was stirred and reacted at 60°C for 90 minutes. The solution was further stirred and reacted at 110°C for 4 hours. The obtained product was filtered, washed with water and was, then, dried at 105°C to obtain a zinc borate (sample A-8). Table 3 shows the chemical composition and properties of the thus obtained zinc borate (sample A-8), Fig. 16 shows an X-ray diffraction image thereof, and Fig. 17 shows an electron microphotograph thereof.

(Comparative Example 1) An aqueous solution was prepared by dissolving 72.5 q of boric acid (B2O3 content of 56.3%) in 1000 ml of pure 15 water. To the aqueous solution were added 95.7 g of a zinc flower (ZnO content of 99.4%) and 217.5 g of boric acid (B₂O₃ content of 56.3%), and were stirred and mixed together such that the molar ratio of B2O3/ZnO was 2.0. Next, the solution was stirred and reacted at 35°C for 120 20 minutes. The solution was further stirred and reacted at 90°C for 4 hours. The obtained product was filtered. washed with water and was, then, dried at 105°C to obtain a zinc borate (sample H-1). Table 3 shows the chemical composition and properties of the thus obtained zinc 25 borate (sample H-1), Fig. 18 shows an X-ray diffraction image thereof, and Fig. 19 shows an electron microphotograph thereof. From Fig. 18, the obtained zinc borate (sample H-1) was the one of the 2.3 type, but was in the form of mixed crystals of a heptahydrate 30 ($2ZnO \cdot 3B_2O_3 \cdot 7H_2O$) and a 3.5 type zinc borate (3ZnO·5B₂O₃·14H₂O), exhibiting a peak due to unreacted zinc oxide. The 2.3 type zinc borate was not obtained in a pure form.

(Comparative Example 2)

35 An aqueous solution was prepared by dissolving 72.5 g

of boric soid (\$203 content of 56.3%) in 1000 ml of pure solve to the someone solve to were solve of the source solve to were solve to the source solve to were solve to the source solve to were solve to the source solve to the solve to water. To the aqueous solution were added 95.7 g of both can be solution of 99.48 and 211.5 g of both can be solution of 99.48 and were attread and mixed water. To the solution of 54.48 and were attread and mixed water. The content of 54.48 and were attread and mixed water. The content of 54.48 and were attread and mixed water. The content of 54.48 and were attread and mixed water attreads attreads and mixed water attreads attreads and mixed water attreads zinc flower (4500 content of 99.4%) and 211.5 9 of boric at the follower (4500 content of 56.3%) and were stirred and mixed and flower such that the molar ratio of 80.7500 was 2.0. acid (18,0) content of 56,3%), and were stirred and mixed of 8,0) from the the molar reacted at for content of 56,3%, and together such that the notar ratio of \$1,00\fm,00 was 2.0. 90 together such that the notar ratio of \$1,00\fm,00 and reacted at 60°C for some the solution was further stirred and reacted at the solution was stirred SOLUTION WE SELECTED AND FERTILES AT 150°C FOT 90 at 150 a minutes. The solution was further stirred and reacted to the solution was further product was filtered, me solution was further dried at 105°C to one; then, drie 150°C for 4 hours. The obtained product was filtered, was the chemical product was the chemical and was. Table 3 shows the chemical product was the chemical and was. a zinc borate (semple H-2); Table 3 shows the chemica of the thus obtains, recommendation and properties of the thus obtains a supplied to the chemical obtains and the chemical obtains a supplied to the chemical obtains and the chemical obtains a supplied to the chemical obtains and the chemical obtains and the chemical obtains a supplied to the chemical obtains and the chemical obtains a supplied to the chemical obtains and the chemical obtains and the chemical obtains a supplied to the chemical obtains a composition and properties of the thus obtained zinc to the thus obtained zinc thus zinc the thus obtained zinc the thus zinc the thus zinc borate (sample R-2); Fig. 20 shows an electron to the state of the sample and Fig. 21 shows an electron and the sample and Fig. 21 shows an electron to the sample and the Washed Will Wares and Wash minutes. Like in Example 1. Table 3 shows chemical finds of zinc to the find of the find of the fine for the fine fore compositions and physical properties of two kinds of compositions and physical properties and commenced by a commenced by a composition of the com borates, 1.e., a commercially available zinc borate to comment of the comment of (eample H-3) manufactured by company A and a commercially by company A and a commercially and by company A and a commercially according to the company of the company and a commercially and a commercially a company a compa microphotograph thereot, and 4) microphotograph thereof. available zinc borate (sample K-4) manufactured by company as a since borate (sample K-4) and X-ray diffraction and Fig. 23 (K-3) shows an electron as Fig. 23 (K-3) shows an electron are fig. 2. (K-3) shows an electron are fig. 3. (K-3) shows are The evaluated results of the Products of the testing the evaluated results of the Products of the testing in devail. The testing the described in devail. Volume resistivity testing (V.R). in a 3.5-inch roll was reasend at 170°C and was reasend at 170°C at 160°C for 7 minutes. and was reasend at 170°C at 160°C for 7 minutes. microphotograph thereof. The following blend 1 was kneeded in a 3.5-inch so a fairthmens a minutes, a sheet having a thickness a sill at 160° for 1 minutes, a sheet having a thickness a sill at 160° for 1 prepare a sheet having a thickness a sill at 160° for 1 prepare a sheet having a thickness a sill at 160° for 1 prepare a sheet having a thickness a sheet having a sheet h mill at 160°C for 7 minutes, and was pressed at 170°C of minutes, and was pressed at hickness of allowers to prepare a sheet having a thickness of mill at 160°C for 7 minutes, and was pressed at 170°C of minutes, and was pressed at 1 invention with now be described below. Mecroons were as nesception nearly (A.S.). The sheet was neasured for its volume resistivity The sheet was measured for its volume resistivity under the sheet was measured for the method stipulated under (chase-cm) in compliance with the method stipulated of the compliance with the method stipulated under the sheet was measured for its work of the compliance with the method stipulated under the compliance with the compliance with the compliance with the method stipulated under the compliance with the compliance with the compliance with the complianc JIS K6723-6.8. Blend 1:

Vinyl chloride resin (P = 1300) 100 parts
DINP (diisononyl phthalate) 50 parts
Tribasic lead sulfate 3.0 parts
Lead stearate 0.5 parts
Sample 5 to 15 parts

(2) Limiting oxygen index (LOI).

The following blend 2 was kneaded in a 3.5-inch roll mill at $160\,^{\circ}\text{C}$ for 7 minutes, and was pressed at $170\,^{\circ}\text{C}$ under 150 kg/cm² to prepare a sheet having a thickness of 1 mm.

The sheet was measured for its limiting oxygen index (LOI value %) in compliance with the method stipulated under JIS K7201B by using a candle combustion tester manufactured by Toyo Seiki Mfg. Co. to evaluate the flame-

Blend 2:

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Vinyl chloride resin (P = 1300)	100 parts
DINP (diisononyl phthalate)	50 parts
Tribasic lead sulfate	3.0 parts
Lead stearate	0.5 parts
Antimony trioxide	0 to 10 parts
Zinc stannate	0 to 10 parts
Sample	1 to 15 parts

Applied examples of the invention will now be

25 described.

(Examples 9 to 17)

Table 4 below shows the results of the volume resistivity testing of the sample A-1 alone, and of the limiting oxygen index testing of when the sample A-1 and the antimony trioxide or the zinc stannate are used in combination.

(Examples 18 to 32)

In the blends 3 and 4 described below, the sample A-1, commercially available magnesium hydroxide,

35 hydrotalcite, polyhydric alcohol, calcium hydroxide,

calcium silicate' epoxy compound and fatty acid salt were calcium silicate' a 7-liter magnetic not sill at ratios introduced into a 7-liter magnetic not sill at ratios calcium silicate; epoxy compound and fathy acid salt we calcium silicate; epoxy compound and fathy acid a remarking the calcium silicate; epoxy compound and fathy acid as remarking the calcium silicate; epoxy compound and fathy acid server ac memory in the calcium silicate; epoxy compound and fathy acid server ac memory in the calcium silicate; epoxy compound and fathy acid server ac memory in the calcium silicate; epoxy compound and fathy acid server ac memory in the calcium silicate; epoxy compound and fathy acid server ac memory in the calcium silicate; epoxy compound and fathy acid server ac memory in the calcium silicate; epoxy compound and fathy acid server accompound acid se introduced into a 7-little magnetic pot mill at ratios and when orong introduced into a 5 together with 2.5 little and when orong shown in rable 5 together from 10 to 20 mm. and when orong have a real and when a real and a shown in Table 5 together with 2.5 liters of magnetic ground in Table 5 together with 2.0 mm, and were shown on twent and by an blown in Table 5 together with 20 20 mm, and were then only weri and by an and were then only weri and by an and were then only weri and by an and any well for and mixed for any mixe balls of dismeters of from 10 to 20 mm, and were ground.

The property of from 10 to 20 mm, and were ground.

The property of from 10 to 20 mm, and were then pulyerized by an and were then pulyerized by an and were then pulyerized by an analysis of for 5 mours, and were the property of Table 5 shows and mixed for 5 hours, and were then pulverial and mixed for 5 hours, and about 100 mmm index to a somption of the state o atomizer to obtain about 300 9 of a sample testing.

atomizer to obtain about 300 9 of a sample testing. parative Examples 33 to 43) at blending retios shown in more many and anomal and anomal anoma Test Pieces were Prepared at Dending retion of when I me the when index tention of when I man owner index tention of when I make the conditions described the conditions described the following the conditions of the limit in owner index tention of when I make in our whole index the limit in owner index tention of the limit index tention of the The resultions described above. The resulting of when index the conditions described above in the resulting on the index that in the conditions are under the conditions are under the conditions are under the conditions are under the conditions and when the conditions are under the conditions and when the conditions are under the conditions are under the conditions and when the conditions are under the conditions are u one resulting of the transcript of 33 to 43) are those of the limiting oxygen index testing of are those of the limiting oxygen are used in archimony trioxide and zinc stamate are used in Table 7 below shows the results of the volume and of the results of the roomarative comparative resistivity testing of the testing of the testing of the testing of the limiting owner index testing of the limiting owner. resistivity testing of the comparative sample H-3, and of the comparative sample the of the comparative sample that the sample sampl the limiting only had an the anthony trioxide of the samples H-1, H-3, H-4 and the anthony trioxide of the samples H-1, H-3, H-4 and in combination. Comparation. Examples 5 to 14) combination. beles 44 to 50 and Comperative Example 15) works a 100 parts by weight of a commission where in morning a come and compensations of the commission where in morning a commission where it may be a commission where it morning a commission where it is not considered to the commission where the commission which is not considered to the commission where the commission while the commission whis (MUC-9023) was inlended at 105°C for 5 minutes to orenare
the himnes were knewded at 105°C for 5 minutes to orenare (Examples 44 to 50 and comparative example arim semples H-1, H-3, H-4 and the antimony and the semples H-1, H-3, H-4 and in combination. (MUC-9025), was blended with samples shown in Table B, and the blended with samples shown in Table B, and the blended with 105°C for 5 minutes to prepare the blended were teated for their wolume anests were teated for their wolume sheets. de were kneeded at 105°C for 5 minutes to Prepare to provide the transition of the control of th sheets, with and in rable 9.

The obtained sheets were tested for their wolfmers are a sheets with the are are a sheet with the are are a shee there are also were as shown in Table 9. For comparison, there are all were as shown in Table 9. For comparison from of them.

The results of a blank blended with none of the results of a blank blended with promotion 16 to 18.

The results of a blank blended with none 16 to 18. shown the results of a blank blended with none of them.

Shown the results of a blank blended Examples 16 to 18)

Shown the results of a comparative Examples 16 to 18)

Shown the results of a constant blended Examples 51 and 52 and comparative No. 3 was welched (Examples 51 and 52 and comparative No. 3 was welched (Examples 51 and 52 and comparative No. 3 was welched (Examples 51 and 52 and comparative No. 3 was welched (Examples 51 and 52 and comparative No. 3 was welched (Examples 51 and 52 and comparative No. 3 was welched (Examples 51 and 52 and comparative No. 3 was welched (Examples 51 and 52 and comparative No. 3 was welched (Examples 51 and 52 and comparative No. 3 was welched (Examples 51 and 52 and comparative No. 3 was welched (Examples 51 and 52 and comparative No. 3 was welched (Examples 51 and 52 and comparative No. 3 was welched (Examples 51 and 52 and comparative No. 3 was welched (Examples 51 and 52 and aples 51 and 52 and Comparative Examples 16 to 18)

100 Gramm of a sodium silicate which were introduced

100 Gramm of a sodium silicate which were introduced

30 Westwylene container, and into which were introduced 100 Grams of a sodium silicate No. 3 were introduced into which were introduced a polyethylene container:

All of And. as commerse; we applied the sample All of Adams. Mete as shown in taple 6. 30 9 of the sample Art or A-4 and as Comparative to each street and the times to boil action was liable will example to mixture was liable to the sample action of the sample action was liable to the sample action was liable to street and the times are sample action. a polyethylene container; and into which were intera polyethylene container; and into which were interned as polyethylene container; and of the since of the si in 30 g measured until the misture was dispersed in the tree in the misture was dispersed in the year. were measured until the mixture was cured.

were as shown in Table 8.

The dispersion property was observed by eyes until the mixture was cured, and was evaluated to be as follows:

- O: Dispersed very well.
- O: Dispersed well.
 - X: Poorly dispersed causing the mixture to be separated or coagulated.

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Table

Comp. Ex.4	H-4	,	•	1 -	58.2	52.5	55.4	169274	m	230	1.49	1.67
Comp. Ex.3	н-3	1.	1	•	55.1	50.1	51.1	141062	8.6	348	1.51	1.71
Comp. Ex.2	H-2	ou	09	150	55.3	51.1	66.5	187918	3.2	17	1.50	1.72
Comp. Ex.1	Н-1	ou	35	90					4.3	25	1.40	2.35
Ex.8	A-8	ou	09	110	82	56.3	76.1	351323	3.1	16	1.51	1.61
Ex.7	A-7	yes	55	75	63.3	46.1	88.0	256795	2.5	13	1.46	1.65
Ex.6	A-6	ou	65	9.0	73.5	40.3	86.1	255033	m	15	1.49	1.61
Ex. 5	A-5	ou	9	85	92.6	71.0	82.2	540432	3.2	16	1.48	1.63
Ex.4	A-4	ou	0.9	80	89.2	42.0	78.3	293343	3.4	16	1.49	1.62
Ex. 3	A-3	ou	45	90	78.9	0.09	70.7	334694	2.9	18	1.47	1.63
Ex.2	A-2	yes	09	06	0.06	59.1	73.5	390947 334694 293343 540432 255033 256795	2.7	20	1.50	1.61
Ex.1	A-1	9	09	90	9.19	64.1	64.5	279489	2.8	15	1.49	1.60
	Sample name	Seed	Synthesizing temp. (1St step) (°C)	Synthesizing temp. (2Nd step) (°C)	Crystallite size (020)	Crystallite size (101)	Crystallite size (200)	Product of crystal- lite sizes (nm3)	Median diameter (µm)	Na (ppm)	Mole ratio (B ₂ O ₃ /ZnO) 1.49	mole ratio (H ₂ O/ZnO)

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Table 4

(Examples using antimony trioxide and zinc stannate in combination)

	Ex.9	Ex. 10	Ex. 11	Ex. 12	Ex. 13	Ex.14	Ex.15	Ex. 16	Ex.17	Unit
PVC (P=1300)	100	100	100	100	100	100	100	100	100	phr
DINE	20	20	20	20	20	20	20	20	20	phr
Tribasic lead sulfate	m	ю	м	m	т	ю	т	т	т	phr
Lead stearate	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	phr
Sample (A-1)	10	ĸ	S	m	10	ъ	വ	М	2	phr
Sb ₂ O ₃		10	ĸ	7						phr
Zinc hydroxystannate (ZHS)		el			ю	10	'n	7	80	phr
O.I value	25.7	33.0	30.0	30.5	28.2	32.5	28.0	29.2	30.0	
V.R 30°C (x1013 Ωcm)	8.02									

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(Examples of coated products and of changing the amounts)

<u>Test blend</u> PVC(P=1300) DINE Tribasic lead sulfate Lead stearate Sample	i 30) lead st grate	ulfate	100 50 3.0 0.5	phr phr phr phr											
	Ex. 18	ex.18 ex.19 ex.20 ex.21 ex.22 ex.23 ex.24 ex.25 ex.26 ex.27 ex.28 ex.29 ex.30 ex.31 ex.32	Ex.20	Ex.21	Ex.22	Ex. 23	Ex. 24	Ex. 25	Ex.26	Ex.27	Ex.28	Ex.29	Ex.30	Ex.31	Ex.32
Sample name	A-9	A-10	A-10 A-11	A-12	A-13	A-13 A-14	A-15		A-16 A-17 A-18	A-18	A-19	A-20	A-21	A-22	A-23
Sample (A-1)	10.0	9.70	9.70	9.70	9.70	9.70	9.70	9.70	9.50	9.50	9.50	9.50	9.50	9.50	9.50
Mg(OH) ₂		0.30							0.5						
Polyhydric alcohol (dipenta- erythritol)			0.30							0.5					
Hydrotalcite (Alkamizer 1)				0.3							0.5				
ESBO (epoxylated soy bean oil)					0.3							0.5			
Ca(OH)2						0.3							0.5		
Magnesium stearate							0.3							0.5	
Calcium silicate								0.3							0.5

26.3

O.I value

Ex. 43

28.0

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able 6

(Examples of using coated products, amount-changed products, antimony trioxide and zinc stannate in combination)

	••	antimony	antimony trioxide and zinc stannate in combination)	and zir	oc stann	ate in co	moinatio	(u		
(Test blend) PVC (P=1300) DINP Tribasic lead Lead stearate Flame-retardi	st blend) PVC (P=1300) DINP Tribasic lead sulfate Lead stearate Flame-retarding agent		100 phr 50 phr 3.0 phr 0.5 phr 10~13 phr							
	Ex.33	Ex.34	Ex.35	Ex.36	Ex.37	Ex.37 Ex.38	Ex.39	Ex. 40	Ex. 41	Ex. 42
Sample (A-10)	ស	10								
Sample (A-11)			r.							
Sample (A-12)				ហ	10					
Sample (A-16)						m				
Sample (A-17)							2	m		
Sample (A-18)									Ŋ	
Sample (A-19)										т
Sample (A-23)										
Sb203	ហ			ro			5			7
Zinc hydroxy stannate		m	ហ		ю	7		7	Ŋ	
0.I value	30.0	28.2	28.0	30.0	28.1	29.8	30.0	29.8	28.0	30.4

Table 7

(Examples using antimony trioxide and zinc stannate in combination)

	Comp.	Comp.	Comp.	Comp.	Comp.	Comp.	Comp.	Comp. Ex.12	Comp. Ex.13	Comp. Ex.14	Unit
PVC(P=1300)	100	100	100	100	100	100	100	100	100	100	phr
DINP	20	20	20	20	20	20	20	20	20	20	phr
Tribasic lead sulfate	т	m	e	ო	က	٣	ო	ო	т	m	phr
Lead stearate	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	phr
Sample (H-1)		Ŋ	- rv								phr
Sample (H-3)	10			Э	2			S	10		phr
Sample (H-4)						က	2			m	phr
Sb ₂ O ₃		Ŋ		7	Ŋ						phr
Zinc hydroxystannate (ZHS)			75			7	s	r.	٠	7	phr
O.I value	25.4	27.8		30.0	27.0 30.0 29.6 29.0	29.0	27.3		27.5 27.8	28.6	
V.R 30°C (x1013 Ωcm)	4.06										

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hab
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Unit	phr	phr			
Ex.50		40		73.5	0.5
Ex. 49		20		20.5	1.0
Ex. 48		10		19.3	1.0 1.1
Ex. 47	40			23.8	1.0
Ex. 46	20			20.8	1.3
Ex.45	10			19.5	1.5
Comp.	1	ı		18	5.6
	Sample (A-1)	Sample (A-3)		0.I value	V.R 30°C(x1017 Acm)

Table 9

(Curing property (at room temperature) of sodium silicate No. 3)

	Example 51	Exmple 52	Example 51 Exmple 52 Comparative Example 16	Comparative Example 17	Comparative Example 18
Sample name	A-1	A-4	boric acid	ZnO	zinc phosphate
Sodium silicate No.3	100	100	100	100	100
Sample (g)	30	30	30	30	30
Curing time	13 min.	18 min.	Readily cured	not cured	35 hr
Dispersing property	0	0	×	0	×